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लेम्बडा-साइहेलोथ्रिन, टेक्नीकल — विशिष्टि

Indian Standard

LAMBDA-CYHALOTHRIN, TECHNICAL — SPECIFICATION

ICS 65.100

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 1997

Price Group 2

Pesticides Sectional Committee, FAD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Division Council.

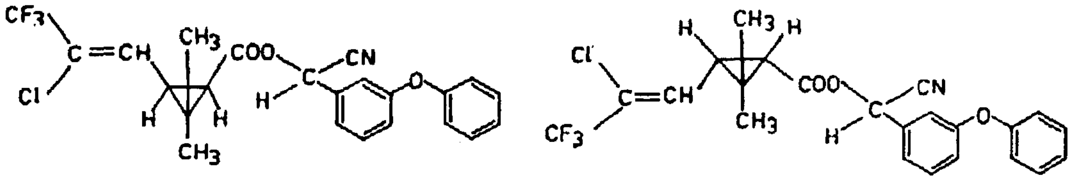
Lambda-cyhalothrin, technical is employed in the preparation of insecticidal formulations. The product is registered under the provisions of *Insecticides Act*, 1968 and Rules framed thereunder.

Lambda-cyhalothrin is the common name of the reaction product comprising equal quantities of (S)-α-cyano-3-phenoxybenzyl (Z)-(1R, 3R)-3-(2-chloro-3, 3, 3-trifluoropropenyl)-2, 2-dimethylcyclopropanecarboxylate and (R)-α-cyano-3-phenoxybenzyl (Z)-(1S, 3S)-3-(2-Chloro-3, 3, 3-tri-fluoropropenyl)-2,2-dimethyl cyclopropanecarboxylate.

Acute oral LD_{50} value of the product for rat male and female per kg body weight is 79 mg and 56 mg respectively. Acute dermal LD_{50} value for rat male and female per kg body weight is 2 165 mg and 1 826 mg respectively.

The empirical and structural formulae and molecular mass of lambda-cyhalothrin are given in Fig. 1 :

Empirical Formula	Molecular Mass
$C_{23}H_{19}ClF_3NO_3$	449.9



(S) (Z)—(1R)— cis —

(R) (Z)—(1S)—cis—

Structural Formula

FIG. 1 STRUCTURAL FORMULA

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the Rules framed thereunder and *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977. However, this standard is subject to the restrictions imposed under these wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

LAMBDA-CYHALOTHRIN, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for lambda-cyhalothrin, technical

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
8190 (Part 1) : 1988	Requirement for packing of pesticides : Part 1 Solid pesticides (*second revision*)
10946 : 1996	Methods for sampling of technical grade pesticides (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a viscous liquid to semi-solid mass. It shall be free from visible extraneous matter.

3.2 The material shall also ,comply with the requirements given in Table 1.

4 PACKING

The material shall be packed in mild steel drums lacquered from inside. It shall also conform to the general requirements given in IS 8190 (Part 1).

5 MARKING

The container shall be marked legibly and indelibly with the following information:

- a) Name of the material;
- b) Name and address of the manufacturer;
- c) Batch or code no;
- d) Date of manufacture;
- e) Date of expiry;
- f) Net mass of contents, kg;
- g) Lambda-cyhalothrin content (percent by mass) as registered under the *Insecticides Act*, 1968 and Rules framed thereunder; and
- h) Any other information required under the *Insecticides Act*, 1968 and Rules framed thereunder and *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977.

Table 1 Requirements for Lambda-Cyhalothrin, Technical
(*Clause* 3.2)

Sl No.	Characteristics	Requirement	Method of Test, Ref to	
			Annex of This Standard	Cl No. of IS 6940
(1)	(2)	(3)	(4)	(5)
i)	Lambda-cyhalothrin content, percent by mass, *Min*	81.0 [*see also* 5(g)]	A	—
ii)	Moisture content, percent by mass, *Max*	0.3	—	4.1
iii)	Acidity (as H_2SO_4), percent by mass, *Max*	0.3	—	11.3.2

5.1 BIS Certification Marking

The product may also be marked with Standard Mark.

5.1.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn according to IS 10946.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in various tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Item* (i)]

DETERMINATION OF LAMBDA-CYHALOTHRIN CONTENT

A-0 Two methods namely Gas Liquid Chromatography (GLC) and High Performance Liquid Chromatography (HPLC) may be used for determination of lambda-cyhalothrin content. However, HPLC may be used as referee method.

A-1 GLC METHOD

A-1.1 Principle

The sample is diluted to appropriate volume with chloroform and lambda-cyhalothrin content is determined by gas chromatography using flame ionization detector and docosane as internal standard.

A-1.2 Apparatus

A-1.2.1 *Syringe* — 10 μl capacity.

A-1.2.2 *Gas Chromatograph*

The apparatus shall be equipped with a flame ionization detector and may be coupled to a printer-plotter-cum-integrator. The operative conditions are suggested below, which can be changed in any other equipment, provided standardization is done :

Column : 50 cm × 2 mm id. Glass packed with 5% OV-101 on Chromosorb WHP(80-100 mesh)

Temperature:
Column oven : 210°C
Injection port : 240°C
Detector : 270°C

Carrier Gas and Flow Rate:
Nitrogen-30 ml per minute

A-1.3 Reagents

A-1.3.1 *Chloroform*

A-1.3.2 *Internal Standard Solution* — Docosane, AR grade.

A-1.3.3 *Lambda-Cyhalothrin Reference Standard* — Known purity.

A-1.4 Procedure

A-1.4.1 *Preparation of Internal Standard Solutions* — Dissolve 225 mg of docosane in 100 ml chloroform.

A-1.4.2 *Preparation of Standard Solution*

Weigh accurately about 0.1g of lambda-cyhalothrin analytical standard into a 50 ml volumertic flask. Add 10 ml of internal standard solution. Dilute with chloroform and shake thoroughly to dissolve lambda-cyhalothrin and make up the volume up to the mark.

A-1.4.3 *Preparation of Sample Solution*

Weigh accurately sufficient sample to contain 0.1 g equivalent of lambda-cyhalothrin and dissolve in about 20 ml of chloroform. Filter the preparation and collect in a 50 ml volumetric flask. Add 10 ml of internal standard solution, dilute with chloroform and mix thoroughly and make up the volume up to the mark.

A-1.4.4 *Estimation*

Inject 2 μl of the standard solution into the gas chromatograph followed by the sample solution with the help of syringe. Measure peak areas of standard and sample solution. Compute the percentage of lambda-cyhalothrin content in the sample.

A-1.5 Calculation

Lambda-cyhalothrin content,

$$\text{percent by mass} = \frac{M_1 \times A_1 \times A_3 \times P}{M_2 \times A_2 \times A_4}$$

where
M_1 = mass in g, of reference standard lambda-cyhalothrin;
M_2 = mass in g, of sample taken for the test;
A_1 = peak area of internal standards in the standard solution injected;
A_2 = peak area of lambda-cyhalothrin in the standard solution injected;
A_3 = peak area of lambda-cyhalothrin in the sample solution injected;
A_4 = peak area of internal standard in the sample solution injected; and
P = percent purity of lambda-cyhalothrin in the reference standard.

A-2 HPLC METHOD

A-2.1 Principle

Lambda-cyhalothrin in the sample is dissolved in hexane and detected by High Performance Liquid Chromatography measuring the absorption at 235 nm and quantitatively estimated by using 2, 6 dinitro toluene as the internal standard.

A-2.2 Apparatus

A-2.2.1 *High Performance Liquid Chromatograph*

A HPLC unit equipped with a ultra-violet (UV) detector and printer-plotter cum integrator and is operated under the following suggestive parameters. These parameters may be varied as per available facilities provided standardization is done:

Column : Stainless steel packed with Partisil silica 5/μ. (25 cm × 4.5 mm id)
Detector : Ultra-violet
Wave length : 235 nm
O.D. Range : 0.16 AUFS
Mobile phase : Hexane : Tetrahydrofuran (99.5 : 0.5)
Solvent flow rate : 1.5 ml per minute
Injection volume : 5 μl

A-2.2.2 *Micro Syringe* — 25 μl capacity.

A-2.3 Reagents

A-2.3.1 *Reference Standard Lambda-Cyhalothrin* — known purity.

A-2.3.2 *Internal Standard* — 2, 6 dinitro toluene.

A-2.3.3 *Hexane* — HPLC grade.

A-2.3.4 *Tetrahydrofuran* — HPLC grade.

A-2.4 Procedure

A-2.4.1 *Preparation of Mobile Phase*

Filter solvents through 0.5 micron membrane filters (millipore type) before use. Mix 995 ml of Hexane and 5 ml of Tetrahydrofuran. Degas before use.

A-2.4.2 *Preparation of Internal Standard Solution*

Dissolve about 3 g of 2, 6 dinitro toluene in 1 litre of hexane.

A-2.4.3 *Preparation of Standard Solution*

Weigh accurately 0.05 g of lambda-cyhalothrin, analytical standard into a 50 ml volumetric flask. Add 10 ml of internal standard solution, dilute to volume with hexane and mix thoroughly to dissolve lambda-cyhalothrin and make up the volume up to the mark. Dilute 10 ml of this solution to 50 ml with hexane.

A-2.4.4 *Preparation of Sample Solution*

Weigh accurately sufficient sample to contain 0.05 g of lambda-cyhalothrin and dissolve in about 50 ml of hexane. Filter the solution into volumetric flask and add 10 ml of internal standard solution. Dilute with hexane and mix thoroughly and make up the volume up to the mark. Dilute 10 ml of this solution to 50 ml with hexane.

A-2.4.5 *Estimation*

A-2.4.5.1 Inject 5 μl standard solution and adjust the HPLC parameter to give complete separation with peak heights half to three forth full scale.

A-2.4.5.2 Proceed with sample analysis by injection of sample solution. Note the areas of lambda-cyhalothrin peak and internal standard peak. Calculate the lambda-cyhalothrin content in the sample.

A-2.5 Calculation

Lambda-cyhalothrin content,

$$\text{percent by mass} = \frac{M_1 \times A_1 \times A_3 \times P}{M_2 \times A_2 \times A_4}$$

where
M_1 = mass in g, of reference standard lambda-cyhalothrin;
M_2 = mass in g, of sample taken for the test;
A_1 = peak area of internal standards in the standard solution injected;
A_2 = peak area of lambda-cyhalothrin in the standard solution injected;
A_3 = peak area of lambda-cyhalothrin in the sample solution injected;
A_4 = peak area of internal standard in the sample solution injected; and
P = percent purity of lambda-cyhalothrin in the reference standard.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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